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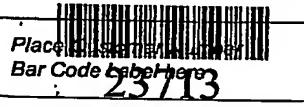
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<input type="checkbox"/> Additional inventors are being named on the _____ separately numbered sheets attached hereto					
TITLE OF THE INVENTION (280 characters max)					
Silica Catalyzed Dissolution of Copper from Sulfur-Containing Copper Minerals					
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<input checked="" type="checkbox"/> Specification Number of Pages 14		<input type="checkbox"/> CD(s), Number 			
<input checked="" type="checkbox"/> Drawing(s) Number of Sheets 2		<input checked="" type="checkbox"/> Other (specify) 			
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Respectfully submitted,

SIGNATURE

Susan K. Doughty

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9/27/02

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116-02P

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P18SMALLREV05

PROVISIONAL APPLICATION FOR LETTERS PATENT

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**SILICA CATALYZED DISSOLUTION OF COPPER FROM SULFUR-
CONTAINING COPPER MINERALS**

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SILICA CATALYZED DISSOLUTION OF COPPER FROM SULFUR-CONTAINING COPPER MINERALS

BACKGROUND OF THE INVENTION

The present invention relates to a cost-effective process for enhanced dissolution of copper from chalcopyrite or other sulfur-containing copper minerals in an acidic oxidative leaching system in the presence of silica and other silica-based compounds.

Chalcopyrite is the most abundant and important source of copper metal available in the earth's crust [Fathi, 1978]. It contains nearly equal parts of copper, iron, and sulfur. The chemical formula is generally written as CuFeS_2 , but it is also represented as $\text{Cu}_2\text{S} \cdot \text{Fe}_2\text{S}_3$, since copper is mainly in a cuprous state and iron in a ferric state. It is found with many sulfide minerals of magnetic origin. It is seen in the metalliferous veins of igneous rocks and in sediments. It may also occur in the secondary enrichment zones of many mineral deposits.

A single crystal of chalcopyrite behaves like a typical semiconductor with a conductivity of 8 to 20 $\text{ohm}^{-1} \text{cm}^{-1}$. It is antiferromagnetic with a hardness of 3.5 to 4.0 on the Mohr scale. The specific gravity is around 4.2. It is closely related to bornite, Cu_5FeS_4 , idaite, Cu_5FeS_6 , and cubanite, CuFe_2S_3 .

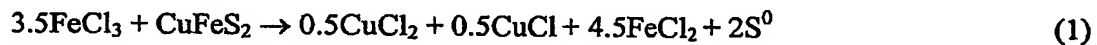
Crystal Structure

The crystal structure of chalcopyrite is tetragonal and it is approximately twice the size of sphalerite. Each metal atom (copper and iron) is coordinated by a tetrahedron of sulfur atoms, and the sulfur atom by a tetrahedron of two copper and two iron atoms. However, the sulfur atom is displaced slightly from the center of the metal tetrahedron, towards the iron-iron edge. The interatomic distance of copper-sulfur is 230.2 pico metre (pm) and iron-sulfur is 225.7 pm. The bonding is essentially covalent in nature with the atoms fluctuating between two ionic states $\text{Cu}^+\text{Fe}^{3+}\text{S}_2^{2-}$ and $\text{Cu}^{2+}\text{Fe}^{3+}\text{S}_2^{2-}$. It is also suggested by Wyckoff [1970] that the resistance of

chalcopyrite to bacterial attack is mainly because of these two ionic states. The first species is more resistant than the second one to bacterial attack.

Leaching and Electrodissolution Studies

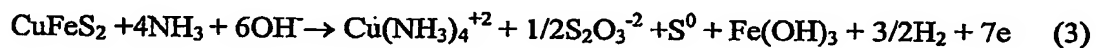
In the usual procedure, the valuable component, copper, is extracted from chalcopyrite by leaching using suitable lixiviants. Widely used lixiviants for chalcopyrite are ferric chloride and ferric sulfate in an acidic medium. The ferric chloride leaching reaction can be written as [Dutrizac, 1978]:



In sulfuric acid [Mateos et al., 1987] containing solutions with ferric sulfate the reaction follows:

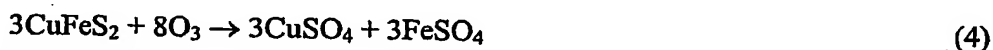


In ammoniacal solution, the reaction is seen to produce thiosulfate and elemental sulfur as below [Reilly and Scott, 1984]:



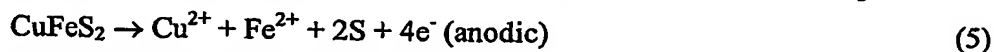
The sulfur layer that forms on the surface of the chalcopyrite in all of the above reactions is passivating and highly protective. Hackl et al. [1995] suggests that this layer is made of copper polysulfide, CuS_n . XPS analysis conducted by Balaz et al. [1996] revealed the existence of sulfur in three different chemical forms: S^{-2} , S^0 , and S^{6+} , when experiments were performed under a combined bacterial and chemical leaching. Other investigators such as Antonijevic et al. [1994] suggest the sulfur to be elemental in nature. Biegler and Swift [1979] observed the properties of sulfur to vary with experimental conditions. When standing over a period of days, sulfur lightened in color due to transformation to the yellow rhombic form. However, there is no real consensus among various investigations as to the nature of the sulfur associated with the passive layer that is formed during chalcopyrite leaching. Nevertheless, it has been established that the decreased leaching of chalcopyrite is due to the passive sulfur layer formation.

Munoz, Miller, and Wadsworth [1979] achieved a maximum copper recovery of about 58% in 20 hours with 4-micron particle size chalcopyrite under drastic conditions of 90° C, 1200 rpm with 1.0 M sulfuric acid. When the particle size was increased to 12 microns, in order to achieve almost the same recovery, leaching had to be performed for 100 hours. When 47-micron particle size was used, it was not possible to achieve more than 20% even after 160 hours. To increase recovery, investigators have tried a number of methods. One of the most successful was developed by Miller et al. [1979] using silver catalysis. In this method, recovery was increased to almost 100%. However, the economics of using silver to extract copper restricted its widespread use. Ozone oxidation was also tried by Halvik and Skrobjan [1990]. The proposed reaction is below:



Again, this process had poor economics and was difficult to commercialize.

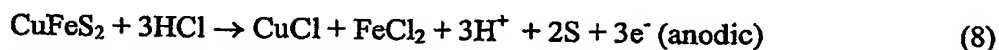
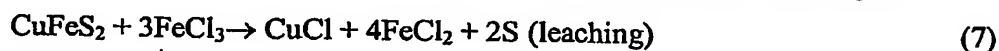
Using high oxidation potential offers a possibility of dissolving chalcopyrite. Chalcopyrite can be made the anode in an aqueous electrolyte with a counter electrode to complete the circuit. The anodic dissolution reaction [Illangovan et al., 1975] can be written as:



And the corresponding cathodic reaction is:



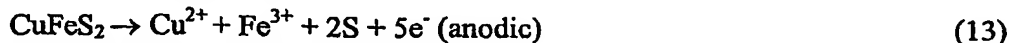
Kruesi et al. [1974] combined chemical leaching with anodic leaching as:



Ferrous chloride solution was electrolyzed separately to get metallic iron. Thus the solution can be regenerated as follows:

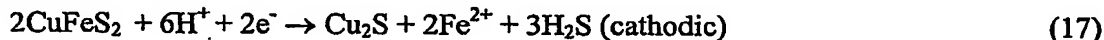


Ilangovan et al. [1975] used a diaphragm made of chlorinated polyvinyl chloride as the anode compartment and a mixture of sulfuric acid and ferric chloride as electrolyte. The reactions found to take place were:



There have been various other studies conducted to dissolve chalcopyrite using an anodic potential, however, the sulfur containing passive layer acts as an insulator between sulfide particles and increases the cell voltage tremendously with the reaction. This is a major problem and with the low conductivity of ores, makes the electrochemical method impractical. The passive layer problem exists in chemical leaching processing also.

The following electrolytic reactions are observed in hydrochloric medium [Biegler et al., 1976]:



From the above equations it can be seen that the products formed are not insulators like sulfur in the anodic process. Therefore, increased cell potential is not a problem. However, the conductivity of the mineral plays a crucial role in the feasibility of this approach.

There is a need in the art for an improved method for leaching of copper from minerals containing copper and sulfur.

BRIEF SUMMARY OF THE INVENTION

The present invention is a method to enhance leaching of copper from sulfur-containing copper minerals using silica-based compounds. Silica-based compounds that are useful in the invention include SiO_2 , silicic acid, fluorosilicic acid, glass sand, borosilicate, dissolved silica, silica gel and colloidal silica. The silica-based compounds may be in any suitable form and any

suitable size, for example finely divided or nanosize. The method is particularly effective where oxidative lixiviants are used in an acidic pH and at a temperature range of 30° to 85° C. This invention is not limited in use to any particular form or size of mineral. The mineral may be in any suitable form, including as found without further processing, crushed or milled.

More particularly, provided is a method of extracting copper from a sulfur-containing copper mineral comprising: adding a lixiviant and a silica-containing compound to a sulfur-containing copper mineral, forming a composition. The method may further comprise adjusting the pH of the composition to be acidic, adjusting the temperature of the composition to between 30 and 85°C, applying light (preferably ultraviolet) to the composition, or any combination. The lixiviant and silica-containing compound may be added to the mineral in any order. The mineral is treated for a sufficient time to extract the desired amount of copper. The composition may be agitated or otherwise treated, as known in the art.

As used herein "lixiviant" is a chemical which leaches copper from a copper-containing mineral. Suitable lixiviants include ferric ion, hydrogen peroxide, chlorate, permanganate, bleach, iodide and bacterial oxidants or a combination thereof. Other standard lixiviants used in the art are also useful in the methods of the invention. Minerals that comprise copper and sulfur may be treated using the methods of the invention. These minerals include chalcopyrite, bornite, chalcosite and others known in the art. "Composition" does not mean a homogeneous solution is formed, merely that the mineral and chemicals are in sufficient contact with each other so that the desired reaction takes place. The composition may further contain components other than those specifically exemplified herein.

BRIEF DESCRIPTION OF THE FIGURES

Figure 1A is a SEM photograph of chalcopyrite surface at pH 1.3 with H₂SO₄ at 0.6V showing the presence of a sulfur layer.

Figure 1B is a SEM photograph of chalcopyrite surface when exposed to 50 % fluorosilicic acid at pH 1.3 with H₂SO₄ at 0.6V, showing the presence of hexagonal silica on the chalcopyrite surface.

Figure 2 is the Pourbaix diagram of the Si-S-H₂O system.

DETAILED DESCRIPTION OF THE INVENTION

The invention may be further understood by reference to the following non-limiting examples.

Example I.

As discussed, the formation of a passive sulfur layer decreases the dissolution of copper from chalcopyrite significantly when leaching is conducted in acidic pH. In order to test that and to get baseline data, chalcopyrite leaching experiments were conducted using ferric as a lixiviant in the absence and presence of different salts. Experimental results are given in Table 1.

Table 1. Leaching of Chalcopyrite with Ferric Lixiviants (-200 mesh size, Temp. 50° C)

Conditions	% Copper Recovery after 15 hrs. Leaching	% Copper Recovery after 72 hrs. Leaching
6 gm/liter chalcopyrite 10 gm/liter ferric-chloride 1.3 pH using H ₂ SO ₄	8.4	14.5
6 gm/liter chalcopyrite 10 gm/liter ferric-chloride 8 gm/liter Thiourea 1.3 pH using H ₂ SO ₄	10.7	19.1
6 gm/liter chalcopyrite 10 gm/liter ferric-chloride 8 gm/liter Thiosulfate 1.3 pH using H ₂ SO ₄	12.6	14

Ferric chloride leaching for 72 hours showed 14% copper dissolution. Addition of thiosulfate and thiourea did not enhance copper recovery.

Example II. Effect of Selected Oxidants

Experiments were conducted in a manner similar to that described in Example I. In this case some strong oxidants were used to destroy the sulfur which would increase the dissolution of copper from chalcopyrite.

Table 2. Effect of Selected Oxidants on the Leaching of Chalcopyrite (-200 mesh size, Temp. 50° C)

Conditions	% Copper Recovery after 15 hrs. Leaching	% Copper Recovery after 72 hrs. Leaching
6 gm/liter chalcopyrite 10 gm/liter ferric-chloride 1.3 pH using H ₂ SO ₄	8.4	14.5
6 gm/liter chalcopyrite 10 gm/liter ferric-chloride 20% commercial bleach (75 gm) 1.3 pH using H ₂ SO ₄	100	100
6 gm/liter chalcopyrite 10 gm/liter ferric-chloride 75 gm/liter chlorate 1.3 pH using H ₂ SO ₄	50	100

These experiments show that a high concentration of oxidants is required to destroy the sulfur layer and thereby enhance the copper dissolution. However, high reagent demand limits the feasibility of this process.

Example III. Effect of Silica During Leaching

In the literature it has been shown that addition of silver can enhance copper leaching. The enhanced leaching is due to the fact that silver sulfide does increase the conductivity and electron transfer. In this investigation selected silica compounds were tested to see if they could be used to increase copper leaching without strong oxidants.

Table 3. Effect of Silica Compounds on Dissolution of Copper from Chalcopyrite (-200 mesh size, Temp. 50° C)

Conditions	% Copper Recovery after 15 hrs. Leaching	% Copper Recovery after 72 hrs. Leaching
6 gm/liter chalcopryrite 10 gm/liter ferric-chloride 1.3 pH using H ₂ SO ₄	8.4	14.5
6 gm/liter chalcopryrite 10 gm/liter ferric-chloride silicic acid (10 cc) 1.2 pH using H ₂ SO ₄	39	81
6 gm/liter chalcopryrite 10 gm/liter ferric-chloride 10 gm SiO ₂ (nanosize) 1.3 pH using H ₂ SO ₄	40	73
6 gm/liter chalcopryrite 10 gm/liter ferric-chloride 10 gm silica gel 1.3 pH using H ₂ SO ₄	29	78
6 gm/liter chalcopryrite 30% hydrogen peroxide (20 cc) 10 gm SiO ₂ (nanosize) 1.3 pH using H ₂ SO ₄	52	79

In all of the above tests it has been shown that addition of fine silica compounds during chalcopryrite leaching can enhance the copper dissolution.

Example IV. Effect of Ultraviolet vs Visible Light

In another series of experiments, the photo-catalyzed nature of silica in enhancing copper dissolution from chalcopryrite was monitored. In this case nanosize silica compounds were added to the chalcopryrite slurry at pH 1.5 in the presence and absence of light. In addition, the effect of ultraviolet light on leaching was determined.

Table 4. Effect of Ultraviolet and Visible Light on

Chalcopyrite Leaching (-200 mesh size, Temp. 50° C)

Conditions	% Copper Recovery after 72 hrs. Leaching
6 gm/liter chalcopyrite 50 cc H ₂ O ₂ (30% strength) 10 gm SiO ₂ (nanosize) 1.3 pH using H ₂ SO ₄ light – visible	78
6 gm/liter chalcopyrite 50 cc H ₂ O ₂ (30% strength) 10 gm SiO ₂ (nanosize) 1.3 pH using H ₂ SO ₄ UV - light – 72 hours	80
6 gm/liter chalcopyrite 10 gm/liter ferric-chloride 10 cc of silicic acid 1.3 pH using H ₂ SO ₄ UV light – 72 hours	88

The above tests showed that UV light in the presence of silica catalyzes the dissolution of copper from chalcopyrite.

Example V. Characterization

In order to observe the structural difference of the surface product, a Scanning Electron Microphotograph (SEM) of the chalcopyrite electrode was subjected to 0.6 volts at pH 1.8 controlled with H₂SO₄. As can be seen, after 72 hours oxidation leaching, a thin sulfur layer was formed on the chalcopyrite surface (Figures 1A and 1B).

In another experiment, chalcopyrite electrode was exposed to 0.6 volts in the presence of fluorosilicic acid. After 72 hours, the surface of the chalcopyrite was inundated with hexagonal

hydroxy-silica crystals. The surface was not protected by a sulfur layer. A similar nature of the surface was observed with colloidal silica and nanosize SiO_2 .

The Eh and pH diagram (see Figure 2) of the Si-S- H_2O system showed that at pH 1.8, at a potential around 0.5 volts, the most predominant species was $\text{H}_2\text{Si}(\text{OH})$ (silicic acid). It is from ElectroChemical Impedance data and other measurements that enhanced leaching were seen in the presence of silica due to the photo-catalytic behavior of colloidal silica particles.

Although the description above contains many specificities, these should not be construed as limiting the scope of the invention, but as merely providing examples of some of the preferred embodiments. For example, minerals other than chalcopyrite may be used. Also, lixivants other than those specifically exemplified may be used. Conditions other than those specifically exemplified may be used, as known in the art without undue experimentation. All references cited herein are hereby incorporated by reference to the extent not inconsistent with the disclosure herewith.

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CLAIMS:

The invention is further described by the following, non-limiting claims.

1. A method of extracting copper from a copper-containing mineral comprising:
adding a lixiviant and a silica-containing compound to a copper-containing mineral, forming a composition.
2. The method of claim 1, further comprising applying ultraviolet light to the composition.
3. The method of claim 1, further comprising adjusting the pH of the composition to acidic.
4. The method of claim 1, further comprising adjusting the temperature of the composition to between about 30 and 85°C.
5. The method of claim 1, wherein the lixiviant is selected from the group consisting of: ferric ion, hydrogen peroxide, chlorate, permanganate, bleach, iodide and bacteria and mixtures thereof.
6. The method of claim 1, wherein the copper containing mineral is chalcopyrite.
7. The method of claim 1, wherein the mineral further comprises sulfur.
8. A method of extracting copper from a sulfur-containing copper mineral comprising:
adding a lixiviant and a silica-containing compound to a sulfur-containing copper mineral,
forming a composition;
adjusting the pH of the composition to be acidic;
adjusting the temperature of the composition to between about 30 and 85°C.
9. The method of claim 8, further comprising applying ultraviolet light to the composition.

10. The method of claim 8, wherein the lixiviant is selected from the group consisting of: ferric ion, hydrogen peroxide, chlorate, permanganate, bleach, iodide and bacteria and mixtures thereof.
11. The method of claim 8, wherein the sulfur-containing copper mineral is chalcopyrite.

ABSTRACT

Leaching of copper from minerals containing sulfur and copper is hampered by the formation of sulfur on the surface of the mineral during conventional processing. An improved method for extracting copper from a sulfur-containing copper mineral is provided, comprising adding a lixiviant and a silica-containing compound to a sulfur-containing copper mineral.

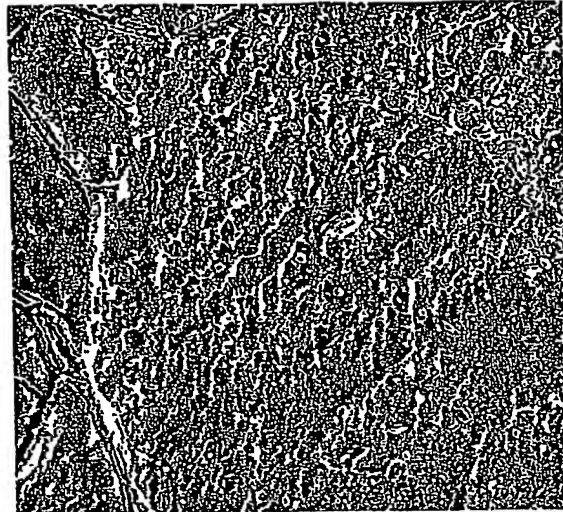


FIG. 1A

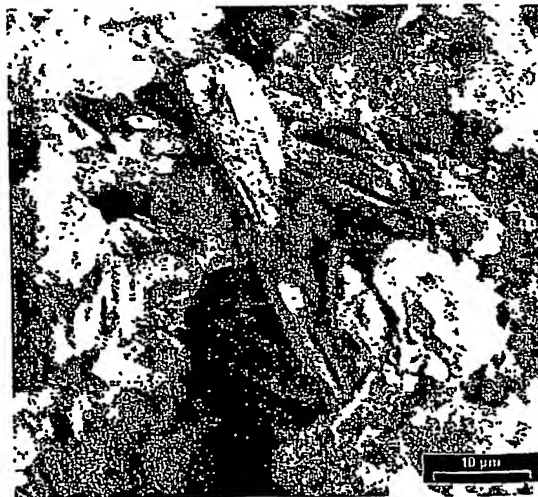


FIG. 1B

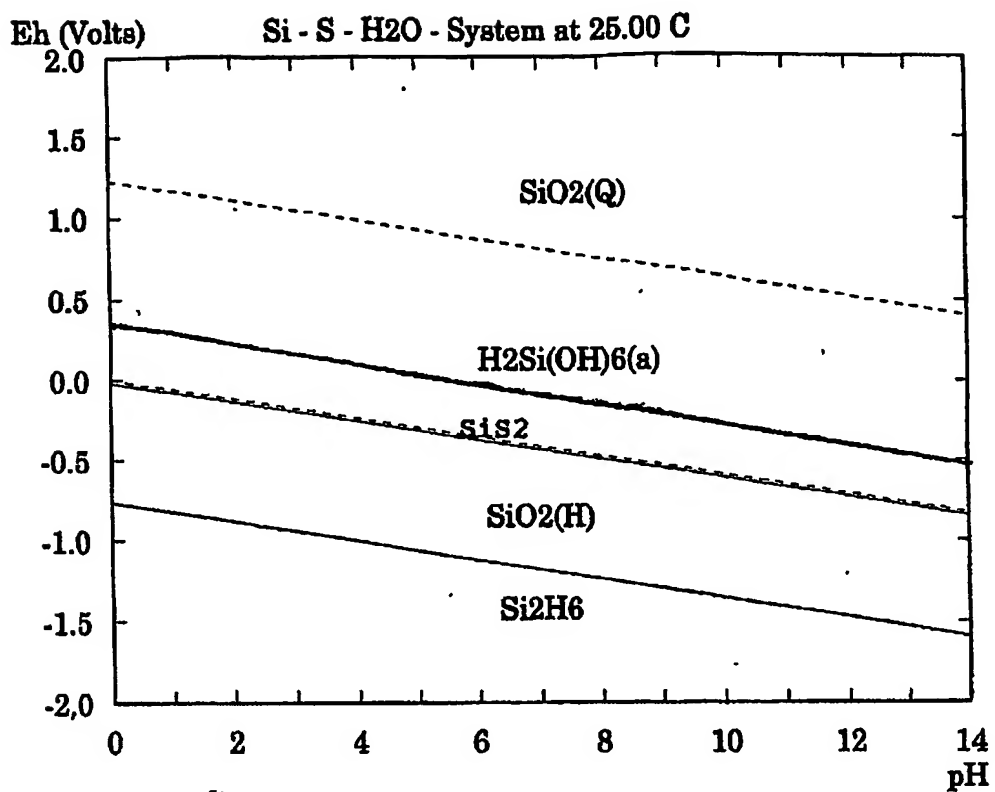


FIG. 2

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